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*Indian Standard*

# DETERMINATION OF WATER IN INSULATING LIQUIDS AND IN OIL-IMPREGNATED PAPER AND PRESSBOARD BY AUTOMATIC COULOMETRIC KARL FISCHER TITRATION — METHOD OF TEST

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NEW DELHI 110002

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**Price Group 4**

Fluids for Electrotechnical Applications Sectional Committee, ETD 03

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Fluids for Electrotechnical Applications Sectional Committee had been approved by the Electrotechnical Division Council.

The test method specified in this standard is a new method, increasingly being used to determine water content in insulating liquids.

This standard has been prepared adopting IEC Document IEC 10 ( Secretariat ) 299 'Determination of Water in Insulating Liquids and in Oil-Impregnated Paper and Pressboard by Automatic Coulometric Karl Fischer Titration' issued by the International Electrotechnical Commission ( IEC ).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

# DETERMINATION OF WATER IN INSULATING LIQUIDS AND IN OIL-IMPREGNATED PAPER AND PRESSBOARD BY AUTOMATIC COULOMETRIC KARL FISCHER TITRATION — METHOD OF TEST

## SECTION 1 DIRECT TITRATION FOR LIQUIDS OF VISCOSITY LESS THAN 100 $mm^2/s$ AT 40°C

### 1 SCOPE

**1.1** This Standard describes a method for the determination of water in unused or used insulating liquids with coulometrically generated Karl Fischer reagent. The method is applicable to water concentrations above 2 mg $kg^1$ in liquids having viscosity less than 100 $mm^2/s$ at 40°C. The precision data given in **11** have been derived for hydrocarbon and ester oils as indicated in **11**.

For liquids of viscosity higher than this value, the preferred method is the high temperature stripping method of Part 2. However, such liquids may also be analysed by the methods of this standard after dilution with a suitable solvent. The precision data of **11** will not be valid for this modified procedure*.

### 2 REFERENCES

**2.1** The following Indian Standards are necessary adjuncts to this standard:

| *IS No.* | *Title* |
|---|---|
| 335 : 1992 | New mineral insulating oils ( *fourth revision* ) |
| 6855 : 1973 | Method of sampling for liquid dielectrics |

### 3 CHEMISTRY

**3.1** The reactions occurring in a Karl Fisher titration are known to be complex, but are essentially of water with iodine, sulphur dioxide, an organic base and an alcohol in an organic solvent. The original Karl Fischer reagent used pyridine and methanol, and the reactions may be expressed as:

$$H_2O + I_2 + SO_2 + 3C_5H_5N \rightarrow 2C_5H_5N.HI + C_5H_5N.SO_3 \qquad (1)$$

$$C_5H_5N.SO_3 + CH_3OH \rightarrow C_5H_5NH.SO_4.CH_3 \qquad (2)$$

Other base-alcohol combinations are possible and may be necessary for titrations on some insulating liquids.

In coulometric Karl Fischer titration the sample is mixed with a base/alcohol solution iodide ion and sulphur dioxide. Iodine is generated electrolytically and reacts with water in a similar way to that shown in reactions (1) and (2).

Iodine is generated in proportion to the quantity of electricity according to Faraday's law, as shown by the following reactions:

$$2I^- - 2\,e \rightarrow I_2 \qquad (3)$$

One mole of iodine reacts with one mole of water stoichiometrically as shown in reactions (1) so that 1 mg of water is equivalent to 10·72 C. Based on this principle it is possible to determine the amount of water directly from the quantity of electricity ( number of coulombs ) required for the electrolysis.

### 4 APPARATUS

#### 4.1 Principle of Operation

Iodine generated by the electrolysis, as shown in reaction (3), reacts with the water in a similar way to the Karl Fischer reactions (1) and (2). The end point of the reaction is detected by a pair of platinum electrodes immersed in the anodic solution. At the end of the titration, excess iodine depolarises the dual platinum electrodes, giving a change in the current/voltage ratio which is used to activate the end-point indicator and to stop the current integrator.

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*Precision for a dilution procedure will be affected by the degree of dilution, which is dependent on the water content of the sample, and the water content and viscosity of the chosen solvent.

The current integrator integrates the current consumed during the electrolysis, calculates the water equivalent according to Faraday's law, and finally displays it in micrograms of water.

### 4.2 Description of the Apparatus

The following description of one suitable form of apparatus is given for illustrative purposes.

The titration vessel has typically the configuration of an electrolysis cell with two compartments connected by a porous diaphragm. The anodic compartment contains a mixture of reagent-solvent and sample ( anodic solution ) the catholic compartment ( generator assembly ) a mixture of reagent and solvent ( cathodic solution ). On both sides of the porous diaphragm are located the electrolysis electrodes.

The black diagram shown in Fig. 1 illustrates the apparatus and includes the following components.

**4.2.1** *Titration Vessel Assembly*

An example of a suitable titration vessel assembly is shown in Fig. 2. However, the changes in instrument technology are such that radically different designs may become available which comply with the technical requirements of this Standard. The exemplified apparatus consists of :

— Flanged glass reaction vessel (a) with sample injection port (b) and drain cock (c).

— Polytetrafluoroethylene lid (d), flanged to match the titration vessel, with three holes to receive the electrodes and drying tube.

— Generator assembly ( combined electrolysis cell ) (e) consisting of a glass tube closed at its lower end by a diaphragm and equipped with platinum electrodes on each side of the diaphragm.

NOTE — The diaphragm may consist of ion exchange membrane, fritted disc, ceramic filter or other system to prevent diffusion of both solutions, while allowing enough current for electrolysis.

— Detector electrodes : dual platinum electrodes for measurement of potential or current (f).

— Polytetrafluoroethylene coated stirrer bar (g).

— Drying tubes (h) to protect the titration vessel and the generator assembly from atmospheric moisture.

— Silicone rubber septa to seal the injection port. It is recommended that crosscuts should be made in the septa before use, to enable blunt, square-ended needles to be used for sample injection ( *see* **5.5** ). Septa should be replaced as required to prevent air leakage as indicated by instrument drift.

**4.2.2** *Detection Circuit*

D.C. constant voltage or a.c. constant current is supplied to the detector electrodes ( dual platinum measuring electrodes ) so that the end point may be detected from the change of the polarised current or voltage.

**4.2.3** *Current Regulator Circuit*

This circuit controls the electrolysis according to the signal from the detector circuit.

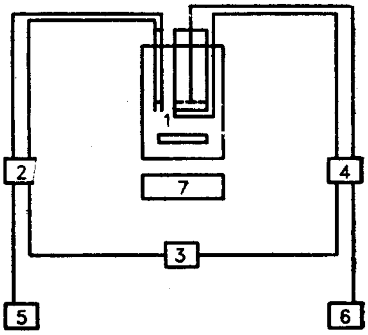
**4.2.4** *D.C. Power Supply for Electrolysis*

**4.2.5** *Endpoint Indicator*

This indicates when the end point has been reached.

**4.2.6** *Current Integrator*

This measures the quantity of electricity consumed by the electrolysis cell during the titration, then calculates and displays the



1 Titration cell assembly
2 Detection circuit
3 Current regulator circuit
4 D.C. Power circuit ( electrolysis )
5 End point displayer
6 Current integrator
7 Electromagnetic stirrer

FIG. 1 BLOCK DIAGRAM OF AUTOMATIC TITRATOR

quantity of water, in micrograms, corresponding to it*.

#### 4.2.7 *Electromagnetic Stirrer*

An electromagnetic stirrer, capable of maintaining a constant speed sufficient to ensure adequate dispersion. ( The content of the titration vessel will not in general be a single phase, since most insulating liquids are not completely miscible with the reagent liquids. )

## 5 REAGENTS AND AUXILIARY MATERIALS

WARNING ! Certain reagents may be detrimental to health and must be handled with proper care.

### 5.1 Reagents

Prepared reagents are commercially available, but care is needed that the reagent is suitable for the particular type of instrument used and the insulating liquid under test. Interfering reactions may take place between methanol based reagents and some silicone compounds. In addition, similar reactions may take place with aldehydes, some ketones, and conjugated unsaturated organic acids, which may be present in oils as degradation products or contaminants. In these cases, reagents not based on methanol are recommended.

Some insulating liquids may require the use of additional or alternative solvents.

**5.2 Desiccant,** for example self indicating silica gel

### 5.3 Lubricant Grease

Polytetrafluoroethylene based or fluorinated hydrocarbon types. Commercially supplied greases to this description have been found satisfactory.

### 5.4 Glass Syringes for Sample Measurement and Introduction

Five ml syringes with needles of suitable length and diameter†. Blunt, square ended needles are preferred, in order to minimise damage to the septa.

## 6 PREPARATION OF THE APPARATUS

Prepare and assemble the apparatus, install the reagents and carry out the stabilisation procedure in accordance with the manufacturer's instructions.

## 7 SAMPLING METHODS

In general, sampling methods are as in IS 6855 : 1973 with following additional recommendations and requirements.

### 7.1 Samples

Although the method is applicable to the analysis of water in any oil sample, the tacking of 'composite' or 'average' samples is not recommended. If samples intended for any other test procedure are used for water analysis, the precision statement of **11** will not be valid, and the results obtained may not be adequately reliable.

### 7.2 Sample Containers

#### 7.2.1 *General Method*

Where it is necessary for samples to be taken for transportation between the location of the equipment and the laboratory, the sample containers shall conform to the following specification:

The samples shall be contained in clean amber coloured glass bottles of nominal capacity 500 ml, having a body height/diameter ratio of not more than 2·0*.

Sample bottles shall be dried by heating in an oven for 16-24 hours at 115°C ± 5°C. While hot, the air content of each bottle shall be displaced by dry nitrogen obtained from a gas cylinder at internal pressure greater than 25 bar, and the bottle capped immediately. After cooling to room temperature, the nitrogen flushing procedure shall be repeated and the bottle re-sealed.

#### 7.2.2 *Samples for Immediate Analysis*

Samples for immediate analysis may be taken directly into dried syringes. The syringes and needles shall be dismantled, dried for at least 8 hours at 115°C ± 3°C, cooled in a desiccator with anhydrous silica gel for a minimum of 2 hours, and kept in the desiccator until required. Before use, the syringe and needle shall be assembled, filled with the oil under test, which is then discharged to waste, and refilled.

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*Some instruments have built-in calculation facilities, and display the water concentration for a specific sample size.

†Needles of length 100 mm and bore 1 mm have been found satisfactory for general use.

* The procedure for drying containers has been found satisfactory for this size and shape of glass bottle. Bottles differing from this will have different surface/volume ratios and may give rise to unacceptable errors through pickup of water from or by the container.

### 7.3 Sampling Procedures

If possible, the sample should be collected from a sampling point fitted with an adjustable valve. The flow shall not be changed during the sampling procedure.

Each sample bottle shall be completely filled and shall be sealed by means of a screw cap with a conical polyolefine insert. After filling, the cap shall be secured by turning with gentle finger pressure only.

If a sampling point is not available, samples may be collected using a clean sampling device such as one of those described in IS 6855 : 1973, other than those constructed of aluminium. The device chosen shall be such that filling of the sample bottle can be achieved rapidly without introducing errors through excessive exposure to air during the transfer.

Sampling devices shall, if possible be dried in the same way as bottles. Devices which are too large for oven drying shall be dried after cleaning by rinsing successively with acetone and hexane followed by dry nitrogen ( as above, *see* **7.2** ) to evaporate traces of solvent.

### 7.4 Storage

Sample for water analysis shall at no time be exposed to direct sunlight. Between the time of collection and analysis, the samples should be kept in the dark, and the time beween collection and analysis shall not in any case be greater than 7 days.

## 8 PROCEDURE

The optimum sample size has been found to be 5 ml for the insulating liquids described in **11**, and for all water contents between 2 and 100 mg $kg^{-1}$. For water contents outside this range, it may be necessary to vary the sample size or to dilute with a suitable solvent. In this case, the precision statements of **11** may not apply.

**8.1** Clean and dry in a well-ventilated oven 115°C ± 5°C, 5 ml glass syringes and needles of a suitable size. Allow them to cool in a desiccator ( *see* **7.2.2** ).

**8.2** Fill a syringe with the insulating liquid, keeping the tip of the needle well below the surface of the liquid. Re-close the glass bottle immediately. Holding the syringe vertically with the needle uppermost, discharge all air bubbles. Discharge the contents of the syringe, which are discarded. Repeat this procedure.

**8.3** Refill the syringe, and align the piston indicator mark with the 5 ml mark on the barrel.

**8.4** Immediately inject the contents of the syringe through the needle inserted through the cross-cut septum. The tip of the needle shall not be allowed to touch the surface of the liquid. Immediately start the electrolysis.

**8.5** Read out the quantity of water titrated ( expressed in $\mu g$ ) from the display when the titration is completed.

**8.6** Carry out a duplicate determination by repeating steps **8.3** to **8.5**.

**8.7** Calculate the mean, $\bar{m}$, of the duplicate determination values.

**8.8** After several determinations, a considerable quantity of insulating liquid may have accumulated. Remove the excess of liquid in accordance with the manufacturer's instructions, which should also be followed in respect of the relative levels of the anode and cathode reagents, after removal of excess insulating liquid, it may be necessary to repeat the procedure for stabilisation of the equipment ( *see* **6** ).

**8.9** Make duplicate determinations of the sample mass of the oil by filling the syringe as in **8.3** and weighing to the nearest 5 mg before and after discharging the oil. Record to the nearest 0·01 g the mean mass M.

## 9 CALCULATION OF THE RESULT

$$\text{Water content} = \bar{m}/M \text{ mg kg}^{-1}$$

where

$\bar{m}$ = mean of displayed readings ( $\mu g$ of water ).

M = mean mass in grams of insulating liquid discharged from 5 ml mark of syringe.

## 10 REPORT

The water content of the insulating liquid sample is the mean of the duplicate determinations, expressed to the nearest integer in mg $kg^{-1}$

## 11 PRECISION

### 11.1 Repeatability

Duplicate results carried out by one operator should be considered suspect at the 95% confidence level if they differ by more than r, where

$r = 0{\cdot}4 + 0{\cdot}019\,c$ ( c = mean of results in mg $kg^{-1}$ )

Each result is the mean of two determinations ( *see* **9** ).

### 11.2 Reproducibility

When two laboratories test different samples of the same insulating liquid, each shall report a result which is the mean of two determinations.

The two means should be considered suspect at the 95% confidence level if they differ by more than R, where

$R = 1{\cdot}7 + 0{\cdot}051\, c$ ( c = mean of results in mg $kg^{-1}$ ).

## SECTION 2 EVAPORATIVE STRIPPING METHOD FOR LIQUIDS OF VISCOSITY GREATER THAN 100 mm²/s AT 40°C

### 12 SCOPE

This standard describes a method for the determination of water in unused or used high viscosity insulating liquids with coulometrically generated Karl Fischer reagent. This method is mainly applicable to water concentration above 2 mg $kg^{-1}$ in liquids having viscosity greater than 100 $mm^2s^{-1}$ at 40°C. However, this method has also been found adequate for the low viscosity liquids, as demonstrated in Annex A.

### 13 OUTLINE OF THE METHOD

A known amount of the insulating liquid is heated in an oven located next to the Karl Fischer apparatus; the water evolved is quantitatively transferred into the titration vessel by a stream of dry nitrogen gas, where coulometric titration is effected.

### 14 APPARATUS

The block diagram of the apparatus is illustrated in Fig. 3.

**Titrator :** Automatic coulometric Karl Fischer ( *see* Fig. 2 ).

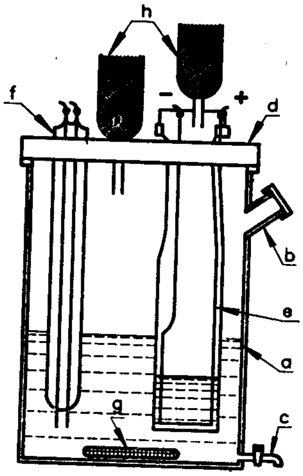
**Evaporator :** Glass vessel, 100 ml capacity; $N_2$ blowing in tube, 1·25 mm inside diameter ( *see* Fig. 4 ).

**Heater :** Clear, transparent electro-conductive glass heater.

**Temperature controller :** Automatic control, accuracy ± 2°C.

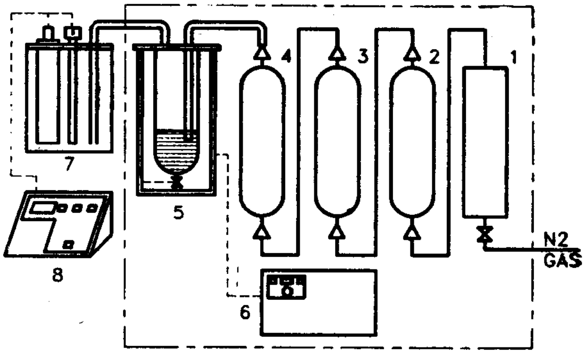
**Carrier gas :** Nitrogen gas ( commercial grade less than 10 $\mu$l/l water ).

**Desiccants for carrier gas:** One silica gel column plus two phosphorus pentoxide columns.



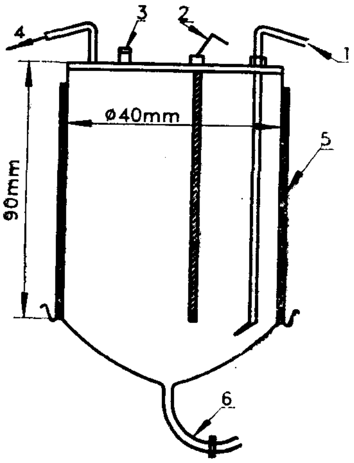
a) Titration vessel ( anodic compartment )
b) Sample injection plug
c) Drain cock
d) Polytetrafluoroethylene lid
e) Generator assembly ( cathodic compartment )
f) Duel platinum for potential measurement electrodes
g) Polytetrafluoroethylene coated stirring bar
h) Drying tubes

FIG. 2 SUITABLE TITRATION VESSEL ASSEMBLY



1 Flowmeter
2 Silica Gel Drying Tubes
3 Phosphorus Pentoxide Drying Tube
4 Phosphorus Pentoxide Drying Tube
5 Evaporator Glass Vessel
6 Temperature Controller
7 Titration Cell
8 Control Unit

FIG. 3 BLOCK DIAGRAM OF AUTOMATIC TITRATOR AND EVAPORATIVE STRIPPING UNIT



1 Nitrogen Inlet Tube
2 Thermocouple
3 Sample Injection Plug
4 Nitrogen + Evaporated Water Outlet Tube
5 Heater
6 Drain Cock

FIG. 4 EVAPORATOR GLASS VESSEL WITH HEATER

## 15 PROCEDURE

**15.1** Operate the Karl Fischer titrator as per procedures specified in Section 1.

**15.2** Inject a sufficient volume ( approximately 10 ml ) of base oil into the evaporator vessel via the septum of the injection plug. This base oil may be the insulating liquid to be tested or any other miscible oil. The purpose of this initial base oil is to immerse the tip of nitrogen inlet tube to permit bubbling of the gas. Purge the whole system with dry nitrogen gas at a flow rate of 200 ml/minute, to thoroughly dry the base oil. Set temperature of the evaporator unit to 130°C ± 5°C. Turn to titration switch on.

**15.3** Allow temperature to stabilize to set value ( approximately 15 minutes ). Continue to titrate to end point. Ignore this reading.

**15.4** Run two blanks of 10 minutes each on this initial base oil by following steps **15.6** and **15.7** of the procedure. If the two blanks are within 5 $\mu$g of water, take the average blank value ( $m_b$ ), if the two blanks differ by more than 5$\mu$g of water, further drying of the base oil may be needed.

**15.5** Inject a suitable quantity of the insulating liquid ( M ) into the evaporator vessel using a syringe ( Minimum needle size = 2 mm inside diameter ). Weigh the syringe before and after liquid injection to obtain the accurate mass of sample. Use a 10 g sample for water contents greater than 10 mg/kg and a 20 ± 5 g sample for water contents less than 10 mg/kg.

**15.6** Turn off titration switch on main unit for a 10 minute period to allow moisture released in the evaporator to accumulate in the titration cell.

**15.7** After 10 minutes, turn on titration switch and allow to titrate to end point.

**15.8** Read the quantity of water titrated ( m ) on the Karl Fischer apparatus.

NOTE — When the water contents are higher than 50 mg kg$^{-1}$ and/or the mass of sample is 20 g, there is possibility that the evaporation of water is not enough during the defined time. In such a case, the perfect evaporation of water by repeating the procedures of **15.6** to **15.7** is recommended.

**15.9** For most insulating liquids, another blank determination should now be made by setting titration switch to off for 10 minutes. If the value determined is close to the original blank value $m_b$ ( that is, within 5 $\mu$g ) no further titration is needed.

## 16 CALCULATION OF WATER CONTENTS

$$\text{Water content} = \frac{\Sigma\,(\,m - m_b\,)}{M}\,\text{mg/kg}$$

where

$m$ = reading on main display ( $\mu$g )
$m_b$ = blank reading ( $\mu$g )
$M$ = mass of insulating liquid tested ( g )

## 17 CARRY OUT A DUPLICATE DETERMINATION

## 18 REPORT

The water content of the insulation liquid sample is expressed as the average of duplicate determinations to the nearest integer mg/kg.

## 19 PRECISION

### 19.1 Repeatability

Duplicate determinations carried out by one operator should be considered suspect at the 95% confidence level if they differ by more than $0{\cdot}60\sqrt{X_m}$ mg/kg ( where $X_m$ is the average of the duplicate determinations ).

### 19.2 Reproducibility

When two laboratories test different samples of the same insulating liquid, each shall produce duplicate results and report their average.

The two averages should be considered suspect at the 95% confidence level if they differ by more than $1.50\sqrt{X_m}$ mg/kg ( where $X_m$ is the average of the two averages ).

## SECTION 3 DETERMINATION OF WATER IN OIL-IMPREGNATED PAPER AND PRESSBOARD

Under Consideration.

## ANNEX A

( *Clause* 12 )

**A-1** Results of comparative determinations of water in polybutenes using direct injection and evaporative stripping.

| Description of Liquid Sample | Viscosity 40°C ( mm²/s ) | Moisture Content, mg/kg | | | | | |
|---|---|---|---|---|---|---|---|
| | | Direct Injection Method | | | Evaporative Stripping | | |
| | | $X_m$ | $( X_1 - X_2 )$ | $0{\cdot}6\sqrt{X_m}$ | $X_m$ | $( X_1 - X_2 )$ | $0{\cdot}6\sqrt{X_m}$ |
| Polybutene ( 015 ) | 700 | 13 | 4 | 2 | 14 | 2 | 2 |
| Polybutene ( 10 ) | 10 000 | Difficult to measure | | | 41 | 0 | 4 |

$X_m$ = Average of duplicate determination $X_1$ and $X_2$.

**A-2** Auther results of comparative determinations of water in different insulating liquids using direct injection and evaporative stripping.

| Description of Liquid Sample | Viscosity at 40°C ( mm²/s ) | Moisture Contents, mg/kg | | | |
|---|---|---|---|---|---|
| | | Direct Injection Method | | Evaporative Stripping | |
| | | X | $\sigma$ | X | $\sigma$ |
| Mineral Oil | 12 | 22 | 0·5 | 19 | 0 |
| Alkylbenzene ( class II ) | 8·5 | 18 | 0·4 | 19 | 0 |
| ( class III ) | 38 | 29 | 0·6 | 28 | 0·5 |
| Alkyldiphenylethane | 6 | 38 | 0·4 | 38 | 0 |
| Alkylnaphthalene | 7 | 12 | 0·5 | 10 | 0 |
| Silicone | 39 | 38 | 0·7 | 36 | 0 |
| Polybutene ( class I ) | 52 | 14 | 0·4 | 15 | 0·5 |
| ( class II type 1 ) | 645 | 20 | 6·5 | 34 | 0·5 |
| ( class II type 2 ) | 28 000 | Impossible to measure | | 16 | 0 |

X = Average of n = 5 $\sigma$ = Standard deviation.

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